Univ of Virginia

Second Semi-Annual Progress Report

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During the second six months of this grant (July 1 - December 31, 1966) about as much progress has been made as was possible in the absence of the CD-ORD spectrophotometer mentioned in the previous report. An instrument will be available to us beginning about April 1, 1967.

1. Apparatus. A photochemical optical bench is presently in operational condition. Problems involved with light sources, monochromatization, heat dissipation, and thermostatting have been resolved. Some quantitative photochemistry has been carried out. The parts of the circular polarizer are partly in hand, but this piece of equipment is not immediately needed.

2. Personnel. A doctoral candidate, Donald E. Schwab, is working one-half time on this project as his thesis research. The principle investigator devotes 15% of his time to the project.

3. Materials. Pure samples of tris(acetylacetonato) complexes of Cr(III), Co(III), and Rh(III) and of tris(8-quinolinol) complexes of Cr(III), Fe(III), and Co(III) have been prepared. A sixteen-foot chromato-

graphic column packed with optically active lactose has been set up and used to elute samples of the tris(acetylacetonato)cobalt(III). This resulted in a partial resolution of the complex, as shown by spectra taken on an ORD instrument at the Arizona State University. Because of the inconvenience of making spectral measurements in this way, the optimal chromatographic conditions were not determined. Further work along these lines will be attempted when a suitable instrument is available.

4. Photochemistry. Since the racemization rates cannot presently be measured, our photochemical work has been confined to irradiation of racemates and to actinometric techniques.

Irradiation of tris(acetylacetonato)cobalt(III) in benzene solution for extended periods of time with a broad band of frequencies has resulted in no chemical change in the complex. This is a particularly helpful situation, since we will have no competing reaction to contend with when observing racemizations.

Mr. Schwab is in the process of testing suitable actinometers for quantum-yield measurements. This is the only work left to be completed before the CD-ORD instrument is needed.

5. Instrumentation. As mentioned in the previous report, the available instruments are not suitable for measuring optical activity of colored samples. The proposed method of adapting the electronic instrument was not successful. Visual instruments are generally not sensitive enough, and the scheme of equipping one with a high intensity light source was abandoned because of the danger of blinding the operator. The principle investigator has negotiated with the Cary Instruments Company to obtain a CD-ORD instrument for a period of one year, beginning about April 1, 1967.

It is hoped that the University will see fit to maintain the availability of the instruments after that period.

New results during the third half-year period will be largely confined to the time that the CD-ORD instrument is operational. The work will probably be concerned with the production of strongly rotating solutions of the metal complexes.